

# इंटरनेट

# मानक

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IS 11720-11 (2001): Methods of Test for Synthetic Rubber, Part 11: Rubber, Raw Styrene- Butadiene - Determination of Soap and Organic-Acid Content [PCD 13: Rubber and Rubber Products]



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“Knowledge is such a treasure which cannot be stolen”



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भारतीय मानक

संश्लेषित रबड़ की परीक्षण विधियाँ

भाग 11 रबड़, कच्चा स्टाइरिन-ब्यूटाडाइन — साबुन और  
कार्बनिक अम्लों का अंश ज्ञात करना

*Indian Standard*

## METHODS OF TEST FOR SYNTHETIC RUBBER

PART 11 RUBBER, RAW STYRENE-BUTADIENE — DETERMINATION OF  
SOAP AND ORGANIC-ACID CONTENT

ICS 83.040.10

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**BUREAU OF INDIAN STANDARDS**

MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG

NEW DELHI 110002

NATIONAL FOREWORD

The Indian Standard ( Part 11 ) which is identical with ISO 7781 : 1996 ‘Rubber, raw styrene-butadiene — Determination of soap and organic-acid content’ issued by the International Organization for Standardization ( ISO ) was adopted by the Bureau of Indian Standards on the recommendation of the Rubber Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

The Committee decided to completely align it with ISO 7781 : 1996 and publish it as a dual number standard. The text of ISO standard has been approved as suitable for publication as Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

- a) Wherever the words ‘International Standard’ appear referring to this standard, they should be read as ‘Indian Standard’.
- b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

In this adopted standard, reference appears to certain International Standards for which Indian Standards also exist. The corresponding Indian Standards which are to be substituted in their place are listed below along with their degree of equivalence for the editions indicated:

<i>International Standard</i>	<i>Corresponding Indian Standard</i>	<i>Degree of Equivalence</i>
ISO 385-1 : 1984 Laboratory glass-ware — Burettes: Part 1 General requirements	IS 1997 : 1982 Burettes ( <i>second revision</i> )	Technically equivalent with minor deviations
ISO 648 : 1977 Laboratory glass-ware — One-mark pipettes	IS 1117 : 1975 One-mark pipettes ( <i>first revision</i> )	do
ISO 1042 : 1983 Laboratory glass-ware — One-mark volumetric flasks	IS 915 : 1975 One-mark volumetric flasks ( <i>first revision</i> )	do
ISO 1795 : 1992 Rubber, raw, natural and synthetic — Sampling and further preparative procedures	IS 5599 : 1999, Rubber, raw, natural and synthetic — Methods of sampling and sample preparation ( <i>first revision</i> )	do

The concerned committee has decided to prepare common methods of test for synthetic rubber under SR ( Synthetic Rubber ) series, namely, IS 11720 and this will be applicable to all types of synthetic rubbers being produced indigenously. The other standards of this series are as follows:

- Part 1 Methods of test for synthetic rubber : Part 1 Determination of antioxidants (SR:1)
- Part 2 Methods of test for synthetic rubber: Part 2 Measurement of vulcanization characteristics with oscillating disc curemeter (SR:2)
- Part 3 Methods of test for synthetic rubber: Part 3 Determination of mooney viscosity
- Part 4 Methods of test for synthetic rubber: Part 4 Determination of volatile matter
- Part 5 Methods of test for synthetic rubber: Part 5 Determination of ash
- Part 6 Methods of test for synthetic rubber: Part 6 Determination of solvent extract
- Part 13 Methods of test for synthetic rubber: Part 13 Determination of gel content

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values ( *revised* )’.

## *Indian Standard*

# METHODS OF TEST FOR SYNTHETIC RUBBER

## PART 11 RUBBER, RAW STYRENE-BUTADIENE — DETERMINATION OF SOAP AND ORGANIC-ACID CONTENT

**WARNING** — Persons using this standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

### 1 Scope

This International Standard specifies a method for the determination of the soap and organic-acid content of raw styrene-butadiene rubber (SBR). The method depends on the extraction of the organic acids and soaps from the rubber by means of a solvent. In practice, therefore, it is convenient to determine both organic-acid and soap contents on separate portions of the same solvent extract. Since the soaps and organic acids present in the rubber are not single chemical compounds, the method gives only an approximative value for the soap and organic-acid content.

The method is applicable to all types of styrene-butadiene rubber, but slight modifications are required for oil-extended rubbers.

### 2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements.*

ISO 648:1977, *Laboratory glassware — One-mark pipettes.*

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks.*

ISO 1795:1992, *Rubber, raw, natural and synthetic — Sampling and further preparative procedures.*

### 3 Principle

A weighed test portion of the rubber, in the form of thin strips, is extracted by ethanol-toluene azeotrope, or ethanol-toluene-water mixture. After making up to standard volume, an aliquot portion of the extract is withdrawn and titrated with standard acid for determination of soap and with standard alkali for the determination of organic acid. With oil-extended rubbers it may be necessary to employ a second aliquot portion of the diluted extract as a control in order to detect the colour change at the end-point.

### 4 Reagents

During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity.

#### 4.1 Ethanol-toluene azeotrope (ETA).

Mix 7 volumes of ethanol with 3 volumes of toluene. Alternatively, mix 7 volumes of commercial grade

ethanol with 3 volumes of toluene, and boil the mixture with anhydrous calcium oxide (quicklime) under reflux for 4 h.

#### 4.2 Ethanol-toluene-water mixture.

Mix 95 cm<sup>3</sup> of ETA (4.1) and 5 cm<sup>3</sup> of water.

#### 4.3 Sodium hydroxide solution,

$c(\text{NaOH}) = 0,1 \text{ mol/dm}^3$ , accurately standardized.

#### 4.4 Thymol blue indicator.

Dissolve 0,06 g of thymol blue in 6,45 cm<sup>3</sup> of 0,02 mol/dm<sup>3</sup> sodium hydroxide solution and dilute to 50 cm<sup>3</sup> with water.

#### 4.5 Hydrochloric acid, $c(\text{HCl}) = 0,05 \text{ mol/dm}^3$ , accurately standardized.

### 5 Apparatus

#### 5.1 Balance, accurate to 1 mg.

#### 5.2 Hotplate.

#### 5.3 Wide-mouthed conical flask, 400 cm<sup>3</sup> to 500 cm<sup>3</sup> nominal capacity.

#### 5.4 Volumetric flask, 250 cm<sup>3</sup>, complying with the requirements of ISO 1042.

#### 5.5 Reflux condenser.

#### 5.6 Conical flask, 250 cm<sup>3</sup>.

NOTE 1 Alternatively, a Soxhlet extractor may be used instead of 5.5 and 5.6.

#### 5.7 Burette, 25 cm<sup>3</sup>, complying with the requirements of ISO 385-1.

#### 5.8 Pipette, 100 cm<sup>3</sup>, complying with the requirements of ISO 648.

#### 5.9 Auto-titrator (optional), only if indicator is not used.

### 6 Procedure for determination of soap content

Sheet out 2 g to 6 g of rubber, selected and prepared in accordance with ISO 1795. Cut into pieces no larger than 2 mm × 2 mm or strips no longer than 10 mm or wider than 5 mm. Weigh a test portion of approximately 2 g to the nearest 1 mg.

Place a circular filter paper in the bottom of the conical flask (5.3) and add 100 cm<sup>3</sup> of ETA extracting solvent (4.1) for all rubbers except alum-coagulated rubbers. For alum-coagulated rubbers, use ethanol-toluene-water mixture (4.2).

Introduce the strips of rubber separately into the flask, swirling after each addition so that the strips are thoroughly wetted with solvent and sticking is minimized.

Fit the reflux condenser (5.5) to the flask (or close the mouth of the flask with a cooling device such as an evaporating dish containing cold water) and boil the solvent very gently under reflux for 1 h.

Transfer the extract to the volumetric flask (5.4), and treat the rubber with a second 100 cm<sup>3</sup> portion of extracting solvent under reflux for 1 h. Add this extract also to the volumetric flask. Rinse the strips with three successive 10 cm<sup>3</sup> portions of extracting solvent, add these washings to the volumetric flask and, after cooling to room temperature, adjust the final volume to 250 cm<sup>3</sup> with solvent.

NOTE 2 Alternatively, the weighed strips of sample may be wrapped in filter paper and placed in a Soxhlet extractor (see note 1) and extracted with ETA (4.1) or ethanol-toluene-water mixture (4.2) under reflux for a minimum of 4 h.

After thorough mixing, pipette 100 cm<sup>3</sup> of the diluted extract into the conical flask (5.6), add six drops of thymol blue indicator (4.4) and titrate the solution with hydrochloric acid solution (4.5) to the first colour change.

If the solution is so dark in colour that the end-point of the titration is likely to be obscure (as may happen with oil-extended rubbers), pipette a second 100 cm<sup>3</sup> into a similar conical flask, add six drops of indicator and use the solution as a colour reference. In comparison, the slight change in colour at the end-point of the titration of the test solution may be more readily observed. The determination of change of colour at the end of the titration can be very difficult in the case of oil-extended SBR. In this case, determination of equivalence point by potentiometry using the auto-titrator (5.9) is more accurate, and is therefore recommended.

Carry out a blank titration on 100 cm<sup>3</sup> of extracting solvent taken from the same stock as was used for the test and using the same indicator as was used for titration of the test portion.

Proceed to 8.1 for calculation of the soap content.

### 7 Procedure for determination of organic-acid content

Proceed exactly as in clause 6 for determination of soap content, but titrate the aliquot portion with so-

dium hydroxide solution (4.3), using thymol blue indicator (4.4).

Proceed to 8.2 for calculation of the organic-acid content.

## 8 Expression of results

### 8.1 Calculate the soap content using the equation

$$w_s = \frac{0,25 \times (V_1 - V_2) \times c_1 \times K_s}{m}$$

where

$w_s$  is the soap content, as a percentage by mass;

$V_1$  is the volume, in cubic centimetres, of hydrochloric acid solution used to titrate the rubber extract;

$V_2$  is the volume, in cubic centimetres, of hydrochloric acid solution used to titrate the blank;

$c_1$  is the actual concentration, in moles per cubic decimetre, of the hydrochloric acid solution (4.5);

$m$  is the mass, in grams, of the test portion;

$K_s$  is the appropriate factor selected from the following:

306 when the soap is to be calculated as sodium stearate,

368 when the soap is to be calculated as sodium rosinate,

337 when the soap is to be calculated as a 50:50 mixture of sodium stearate and sodium rosinate,

322 when the soap is to be calculated as potassium stearate,

384 when the soap is to be calculated as potassium rosinate,

353 when the soap is to be calculated as a 50:50 mixture of potassium stearate and potassium rosinate,

345 when the soap is to be calculated as a 50:50 mixture of sodium stearate and potassium rosinate or of sodium rosinate and potassium stearate.

NOTE 3 Since the soaps present in the rubber are not single chemical compounds, the value assigned to  $K_s$  gives only an approximate value for the soap content. A test for rosin is given in annex A.

### 8.2 Calculate the organic acid content using the equation

$$w_o = \frac{0,25 \times (V_3 - V_4) \times c_2 \times K_o}{m}$$

where

$w_o$  is the organic acid content, expressed as a percentage by mass;

$V_3$  is the volume, in cubic centimetres, of sodium hydroxide solution used to titrate the test solution;

$V_4$  is the volume, in cubic centimetres, of sodium hydroxide solution used to titrate the blank;

$c_2$  is the actual concentration, in moles per cubic decimetre, of the sodium hydroxide solution (4.3);

$m$  is the mass, in grams, of the test portion;

$K_o$  is the appropriate factor selected from the following:

284 when the acid is to be calculated as stearic acid,

346 when the acid is to be calculated as rosin acid,

315 when the acid is to be calculated as a 50:50 mixture of stearic acid and rosin acid.

NOTE 4 Since the organic acids present in the rubber are not single chemical compounds, the value assigned to  $K_o$  gives only an approximate value for the organic acid content. A test for rosin is given in annex A.

## 9 Test report

The test report shall include the following information:

- all details required for identification of the sample;
- a reference to this International Standard;
- the soap and/or the organic-acid content;
- how the soap and/or organic acid was estimated — as stearic acid, mixed sodium stearate and potassium rosinate, etc.;
- the date of the test.



## **Annex A**

(informative)

### **Test for rosin**

#### **A.1 Reagents**

##### **A.1.1 Acetic anhydride.**

##### **A.1.2 Sulfuric acid** solution.

Carefully mix 65 g of sulfuric acid ( $\rho_{20} = 1,84 \text{ g/cm}^3$ ) with 35 g of water.

##### **A.1.3 Potassium permanganate** solution, $c(\text{KMnO}_4) = 0,000 2 \text{ mol/dm}^3$ .

#### **A.2 Procedure**

Mix a small amount of sample with about  $3 \text{ cm}^3$  of acetic anhydride (A.1.1). Add 2 drops of sulfuric acid (A.1.2). The reaction is positive for rosin if a fugitive violet colour appears which, at the moment of its maximum intensity, is stronger than the colour of the potassium permanganate solution.

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards : Monthly Additions'.

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**BUREAU OF INDIAN STANDARDS**

**Headquarters:**

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110 002  
Telephones : 323 01 31, 323 33 75, 323 94 02

Telegrams: Manaksanstha  
( Common to all offices )

**Regional Offices :**

**Telephone**

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg  
NEWDELHI 110 002

{ 323 76 17  
  323 38 41

Eastern : 1/14 C. I. T. Scheme VIIM, V. I. P. Road, Kankurgachi  
CALCUTTA 700 054

{ 337 84 99, 337 85 61  
  337 86 26, 337 91 20

Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160 022

{ 60 38 43  
  60 20 25

Southern : C. I. T. Campus, IV Cross Road, CHENNAI 600 113

{ 235 02 16, 235 04 42  
  235 15 19, 235 23 15

Western : Manakalaya, E9 MIDC, Marol, Andheri (East)  
MUMBAI 400 093

{ 832 92 95, 832 78 58  
  832 78 91, 832 78 92

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